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A Step Towards Multipoint Hydrogen Sensing : Development of Metal Hydride-Coated FBG Hydrogen Sensors

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ABSTRACT

Hydrogen, a key component of a net-carbon free society, requires precise sensing solutions. This research focuses on the development of metal hydride-coated Fibre Bragg Grating (FBG) based hydrogen sensors, marking a significant step towards the realisation of multipoint hydrogen sensing systems—a growing demand in the industry. The performance of three FBG sensors coated with nanometre-thick tantalum, palladium, and palladium-gold hydrogen sensing metal thin films, deposited via magnetron sputtering, is presented. Among these, the novel tantalum sensor exhibited the best performance, achieving a minimum detection limit of 50 ppm and an enhanced sensitivity below 0.1% H₂ levels at room temperature.

Keywords: hydrogen sensors, fibre optic sensors, fibre bragg grating, metal hydrides, tantalum

1. INTRODUCTION

As industries transition away from fossil fuels towards sustainable, carbon-free alternatives, hydrogen has emerged as a pivotal driver of near-zero carbon emission fuels. However, its low explosion limit in air (4%) necessitates precise and efficient detection techniques to ensure safe operation by monitoring potential leakages. Fibre optic sensors have emerged as highly viable candidates for hydrogen detection due to their numerous advantages, including compact size, spark-free operation, reduced susceptibility to electromagnetic interference, high sensitivity, and remote sensing capabilities.¹

Among the various fibre optic hydrogen sensors reported to date,¹ such as surface plasmon resonance based, micro-mirror, and interferometric sensors —grating-based sensors have gained significant traction.² This is primarily due to their ease of fabrication, wavelength-modulated interrogation methods that mitigate source variations, and, importantly, their potential for multiplexing. Fibre Bragg Grating (FBG)-based sensors, in particular, stand out in this regard.² When a hydrogen-sensitive material that can gradually expand (elastically) upon hydrogen interaction, such as a metal hydride, is deposited on an FBG surface, the longitudinal expansion caused by hydrogen absorption can be translated into a measurable strain signal from the FBG to develop a hydrogen sensor. Palladium (Pd) has been extensively explored as a hydrogen sensing material, despite its weaknesses such as hysteresis and delamination challenges.³ The hysteresis of Pd can be mitigated by alloying it with gold (Au), albeit at the expense of significantly reduced optical contrast.⁴ Recently, tantalum (Ta) has also been introduced as a novel and superior hydrogen sensing material, offering improved performance over Pd and its alloys.⁵ This research focuses on comparing the Bragg response of three metal hydride-coated FBG hydrogen sensors— Ta, Pd, and Pd-alloy (Pd_{0.6}Au_{0.4}) across a hydrogen concentration range of 0.005% to 4%, while reporting a Ta based FBG sensor for the first time. Nanometre-thick metal thin films were deposited onto the fibre optic surface using magnetron sputtering. The selected concentration range aligns with the explosion limit of hydrogen in air. The design, fabrication, and performance of these sensors are detailed in the following sections.

2. SENSOR DESIGN AND FABRICATION

In this work, three metal thin-film-coated FBG sensors were developed. For all three sensors, a 4 nm titanium (Ti) layer was first deposited on the FBG surface as an adhesion layer. Subsequently, the sensing layers were applied: a 50 nm Pd layer for the Pd sensor and a 50 nm Pd_{0.6}Au_{0.4} layer for the Pd_{0.6}Au_{0.4} sensor. The novel Ta FBG sensor featured a two-layer stack: a 50 nm Ta thin film serving as the primary sensing element, overlaid with a 15 nm Pd_{0.6}Au_{0.4} capping layer.

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The capping layer had a dual purpose: protecting the Ta layer from oxidation and catalysing the dissociation of hydrogen molecules into individual atoms.⁵ Finally, all three sensors were coated with a 30 nm polytetrafluoroethylene (PTFE) layer to protect against potential contaminants, such as humidity, carbon-dioxide, and carbon-monoxide, which could deactivate the sensors. The sensing metal layer stack can reversibly absorb hydrogen (forming metal-hydrides),⁴ causing longitudinal expansion or contraction of the fibre even at trace hydrogen concentrations. The FBG optical transducer converts these physical changes into a strain signal by altering the Bragg wavelength of the metal-hydride-coated FBG. The magnitude of these changes depends on the longitudinal expansion of the metal hydride layer, which depends on the hydrogen concentration in the surrounding environment. Thus, the shift in the centre wavelength of the Bragg peak can be used to monitor and quantify the hydrogen concentration. This principle forms the basis of the FBG hydrogen sensors developed in this work.

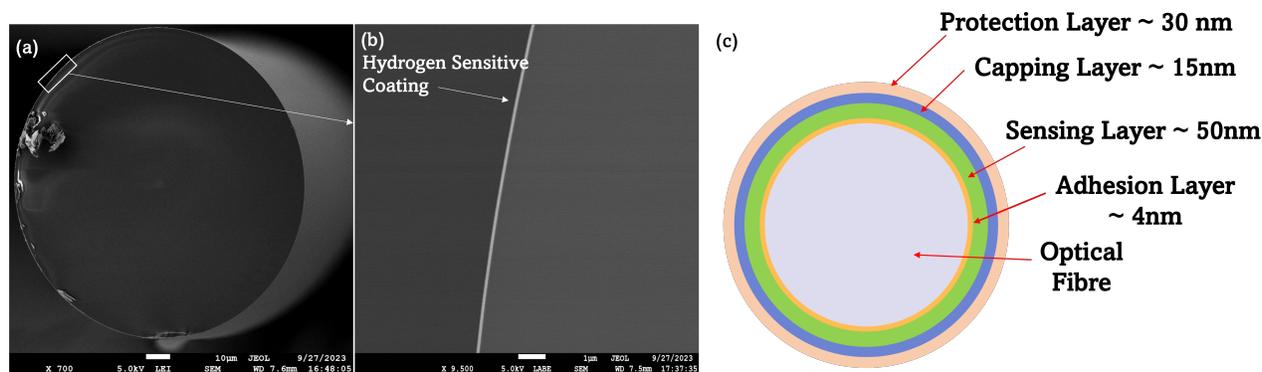


Figure 1: (a) Cross-sectional SEM image of a Ta coated FBG sensor. (b) Zoomed area of the marked white box with the Metal thin-film layer. (c) A schematic of the hydrogen sensing metal thin film stack (Not to scale)

To fabricate the sensors, the fibre optic surface was first cleaned with acetone, and a stack of metal thin-film layers was then deposited onto the FBG surface using magnetron sputtering (AJA International, with a base pressure of 10^{-6} Pa). During deposition, the optical fibre containing the FBG was mounted on a rotating plate under 0.3 Pa of argon (Ar) to ensure uniform coating. The sputtering parameters and conditions followed those detailed in our previous work,⁵ with deposition thickness controlled by adjusting the deposition time. The FBGs used in this work were the type of DTG (Draw Tower Grating) from the company, FBGS. The Bragg wavelengths were selected to fall within the 1460 nm to 1620 nm range, corresponding to the operating wavelength region of the Hyperion si155 FBG interrogator from LUNA Inc. This interrogator offers a dynamic range of 35 dB and a wavelength resolution of 1 pm. Each FBG was 8 mm in length. Figure 1 shows a cross-sectional Scanning Electron Microscopic (SEM) image of a Ta coated FBG sensor. In Figure 1(b), the metal thin-film layer is visible within the marked white boxed area. A schematic of the sensing metal thin film stack is depicted in Figure 1(c).

3. EXPERIMENTAL SETUP

A leak-proof gas chamber was supplied with gas from three cylinders—100% Ar, 4% H₂ in Ar, and 0.1% H₂ in Ar—via three Mass Flow Controllers (MFCs). The MFCs were used to mix and regulate the flow rates from the cylinders, enabling precise control over the hydrogen concentration inside the chamber. The total gas flow into the chamber was maintained at 200 sccm, and the hydrogen concentration was calculated based on the flow ratios from the cylinders. The coated FBG sensors, along with another bare, uncoated FBG (for temperature compensation) were mounted securely inside the chamber and connected to the LUNA si155 interrogator through a fibre optic feedthrough provided by SQS Vláknová Optika A.S. The Bragg wavelength of each of the four FBG sensors was continuously monitored and recorded at a rate of 10 Hz using a moving average of 100 points at room temperature over a range of hydrogen concentrations, from 0.005% to 4% (one complete loading and unloading cycle), to evaluate sensor performance. Each hydrogen concentration step was maintained for 20 minutes. A schematic diagram of the same experimental setup can be found in our previous work.⁶

4. RESULTS AND DISCUSSION

Figure 2 illustrates the temperature-compensated Bragg wavelength shifts of the three metal-hydride-coated FBG hydrogen sensors, alongside the response of the uncoated bare FBG (Figure 2(d)), across hydrogen concentrations ranging from 0.005% to 4%. It is evident that all metal-hydride-coated sensors exhibited stepwise responses to changes in hydrogen concentration, whereas the uncoated FBG responded only to ambient temperature fluctuations within the gas cell.

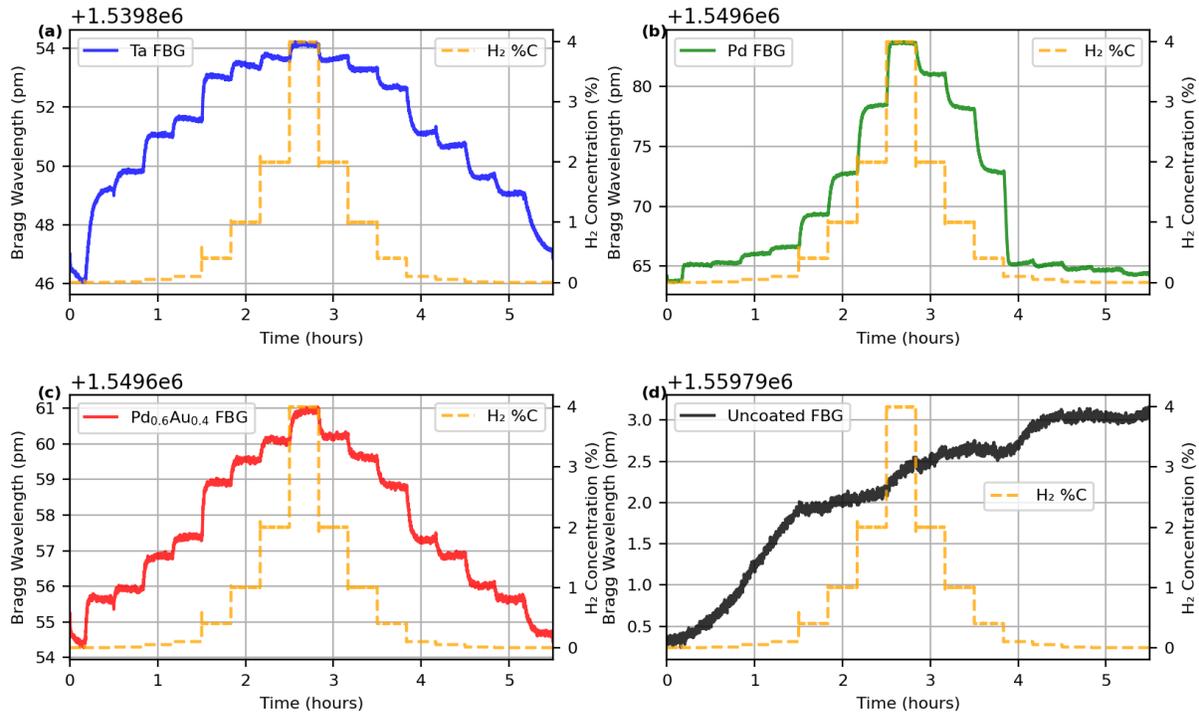


Figure 2: Bragg wavelength shift under varying hydrogen concentration levels; (a) Ta-coated FBG. (b) Pd coated FBG. (c) Pd_{0.6}Au_{0.4} coated FBG. (d) Bare Uncoated FBG.

A key observation is the clear hysteresis in the response of the Pd-coated sensor at hydrogen concentrations above 0.1% (Figure 2(b)). In contrast, Pd_{0.6}Au_{0.4} and Ta-coated sensors displayed negligible hysteresis effects. The hysteresis in the Pd-coated sensor arises from phase transitions occurring at higher hydrogen concentrations.⁴ By alloying Pd with Au, these hysteresis effects are mitigated, as demonstrated by the Pd_{0.6}Au_{0.4} sensor's response. However, Pd exhibited the highest Bragg wavelength shift of 18 pm, compared to the ~5 pm shift observed for the Ta and Pd_{0.6}Au_{0.4} sensors when transitioning from 0.005% to 4% hydrogen levels. Another significant observation is that the Ta and Pd_{0.6}Au_{0.4} coated sensors exhibited higher sensitivity at lower hydrogen concentrations, particularly below 0.1%, making them well-suited for detecting minute hydrogen leaks. This is further supported by the linear sensitivity trend observed on a logarithmic scale of hydrogen concentrations, as shown in Figure 3. Between the Ta and Pd_{0.6}Au_{0.4} sensors, a comparison of the initial shift from 100% Ar (no hydrogen) to 0.005% hydrogen revealed that the Ta sensor exhibited a Bragg wavelength shift of approximately 3 pm, compared to ~1 pm for the Pd_{0.6}Au_{0.4} sensor, underscoring Ta's even superior sensitivity at lower hydrogen concentrations. This is further emphasised by the steeper slope of the Ta response observed in Figure 3 compared to that of the Pd_{0.6}Au_{0.4} sensor. Both the Ta and Pd_{0.6}Au_{0.4} FBG hydrogen sensors demonstrated a minimum detectable H₂ concentration of 50 ppm (0.005%), constrained by the current flow rates of the MFCs used. These promising preliminary results from the Ta and Pd_{0.6}Au_{0.4}-coated FBG sensors highlight their potential for developing FBG based hydrogen sensing systems. Such systems can be easily multiplexed to include multiple sensors on a single optical fibre, enabling multipoint hydrogen detection. Future work will focus on optimising the deposition thickness of the sensing materials to further enhance sensitivity and performance of these metal-hydride-coated FBG hydrogen sensors.

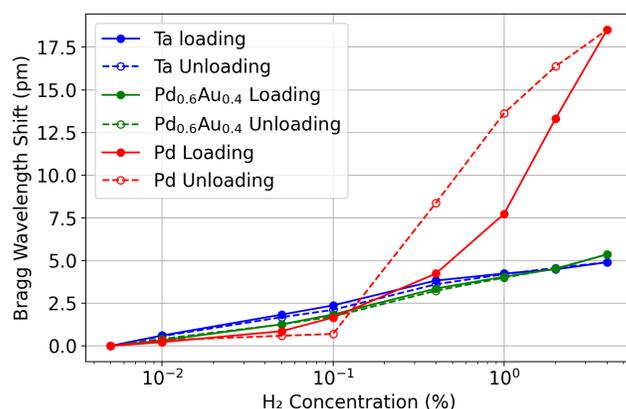


Figure 3: Bragg response comparison of Ta, Pd, and Pd_{0.6}Au_{0.4} FBG sensors at varying hydrogen concentrations.

5. CONCLUSION

Three metal-hydride-coated FBG hydrogen sensors were successfully fabricated using magnetron sputtering to deposit nanometre-thick metal thin films on the fibre optic surface. The Bragg response of these sensors were recorded across hydrogen concentrations ranging from 0.005% to 4%. Both the Ta and Pd_{0.6}Au_{0.4}-coated sensors exhibited a nearly hysteresis-free response and displayed a linear relationship with the logarithm of hydrogen concentration, making them particularly suitable for detecting low hydrogen levels. Both these sensors demonstrated the ability to detect a minimum hydrogen concentration of 50 ppm, which was limited by the concentration achievable with the current flow controllers. In contrast, the Pd-coated sensor demonstrated higher wavelength shifts at concentrations above 0.1% but was significantly affected by hysteresis. Among the three, the novel Ta-coated sensor showed the best performance at low hydrogen concentrations. These promising preliminary results highlight the potential of metal-hydride-coated sensors for developing multipoint hydrogen detection systems integrated on a single fibre.

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